# GLUTARALDEHYDE TREATMENT OF INSOLE LEATHER

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## ABSTRACT

Belly leather and cut insoles were treated with glutaraldehyde to improve perspiration resistance. One percent glutaraldehyde added in the oil wheel during commercial processing markedly increased the resistance. Dipping finished leather pieces for fifteen minutes in a dilute glutaraldehyde solution, to give a similar pickup, also produced good results. Resistance to perspiration was evaluated by determining the relative loss in tensile strength.

## INTRODUCTION

The tanning ability of glutaraldehyde was reported by Fein and Filachione (1) and by Seligsberger and Sadlier (2) in 1957. Exploratory work to establish the most desirable conditions for tanning followed immediately (3). Leather tanned with glutaraldehyde showed remarkable resistance to perspiration (4). Glutaraldehyde can be used alone, simultaneously with chrome or with vegetable tanning agents, or as a retannage with either (5, 6).

In this study glutaraldehyde was used to treat vegetable-tanned insole leather. The purpose of the treatment was to impart perspiration resistance to a low cost item at minimum expenditure. It was added to belly leather in the oil wheel, as the most feasible approach, and to belly pieces and cut insoles by dipping in a dilute solution as an alternative post-treatment.

## EXPERIMENTAL

### Addition of Glutaraldehyde in the Oil Wheel

Two packs of 25 vegetable-tanned bellies were treated with glutaraldehyde in the oil wheel at a co-operating tannery. One-half percent of glutaraldehyde (100 percent basis) based on the wrung weight of the bellies was added to the first pack; one percent of glutaraldehyde (100 percent basis) was added to the second pack. Before the addition of glutaraldehyde four bellies were removed

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from each pack and cut in half. One half of each was returned to the oil wheel, while the other half was processed in the regular way and used as a control.

Eight adjacent tensile strips were cut from each half-belly of the finished leather. Thickness measurements were recorded. The strips were treated with synthetic perspiration solution as described below and prepared for tensile strength tests.

## Addition of Glutaraldehyde by Dipping in a Dilute Solution

Finished belly leather and cut insoles, buffed and full grain, were obtained from individual processors. Approximately eight to ten pieces of leather (4 x 12 inches) cut from each belly or eight to ten insoles (4 x 12 inches) were used in each experiment. The glutaraldehyde used was a commercially available 25 percent aqueous solution. From this a solution containing 2.5 percent glutaraldehyde was prepared. One piece of leather was dipped at a time in ten times its weight of solution. Each piece of leather was soaked in the solution for 15 minutes without agitation. Preliminary tests indicated that under these conditions a 15-minute dip gave sufficient protection.

The percent pickup of glutaraldehyde was calculated by the formula:

$$\% \text{ Pickup} = \frac{(\text{wt. of dipped insole} - \text{wt. of insole before dip}) \times 0.025}{\text{wt. of insole before dip}} \times 100$$

This calculation can be done immediately. The results compared favorably with the percent pickup obtained from the weighing of insoles equilibrated at constant temperature and humidity before and after dip. Equilibration required about ten days.

Tensile strips cut from each piece were treated as described below and prepared for tensile strength determinations.

## The Perspiration Test

Synthetic perspiration solution was made according to Federal Test Method Standard 311, Method 3211, with the addition of 4 M urea (7). Leather strips were treated in the Clarke-Flaharty Insole Tester (8, 9), using the following procedure:

Heat Insole Tester to 36°C.
Mount leather strips on Tester.
Flex 300 cycles dry (approximately five minutes).
Add three ml. of synthetic perspiration solution while running.
Flex 2100 cycles (approximately 30 minutes).
Add two ml. of perspiration solution while running.
Flex 9300–9508 cycles (approximately two hours).
Remove leather strips from instrument.

Heat in oven at 40.5°C. for one hour.
Place in conditioning room for 24 hours.
Repeat test next day.

The insole tester simulates a walking action. It produces flexing under pressure over a 1.5-inch area in the presence of a perspirationlike fluid.

### Tensile Strength

The leather strips were dried at 23°C. and 50 percent R.H. (10) before the tensile strength was determined (11). The strength of the leather decreased with the increased number of flexes. In addition, the further deterioration by perspiration was measured. This effect was determined by the comparative loss in tensile strength between glutaraldehyde-treated and untreated leathers subjected to the same perspiration test. All tensile test pieces used for comparison were cut adjacent to each other wherever possible. Since this was not possible with insoles, a number were selected for control testing.

## RESULTS

Table I indicates that 0.5 percent glutaraldehyde applied in the oil wheel produced very little or no perspiration resistance. One percent glutaraldehyde

TABLE I

TENSILE STRENGTH OF BELLIES TREATED IN OIL WHEEL WITH GLUTARALDEHYDE AND AFTER SUBJECTING TO PERSPIRATION TEST

| | Regular Leather* | | Glutaraldehyde Treated† | | Average Loss | |
|---|---|---|---|---|---|---|
| Belly No. | Control‡ PSI | Treated with Perspiration PSI | Control‡ PSI | Treated with Perspiration PSI | Regular Leather % | Glutaraldehyde Treated % |
| | | | 0.5%** | | | |
| 1 | 4050 | 1900 | 3870 | 2170 | 53.0 | 43.9 |
| 2 | 4330 | 1510 | 4310 | 1650 | 65.1 | 61.7 |
| 3 | 4460 | 1490 | 5180 | 1940 | 66.6 | 62.5 |
| 4 | 4150 | 1410 | 4600 | 1530 | 66.2 | 66.8 |
| Average | 4250 | 1580 | 4490 | 1820 | 62.7 | 58.7 |
| | | | 1.0%** | | | |
| 1 | 4340 | 1870 | 4190 | 3120 | 56.9 | 25.6 |
| 2 | 5210 | 1640 | 4090 | 3060 | 68.5 | 25.2 |
| 3 | 4110 | 1620 | 3670 | 3110 | 60.7 | 15.3 |
| 4 | 3630 | 1020 | 4090 | 2690 | 71.9 | 34.2 |
| Average | 4320 | 1550 | 4010 | 3000 | 64.5 | 25.1 |

*Half-belly processed in regular way.
†Half-belly treated with glutaraldehyde.
‡Control represents specimens not subjected to perspiration.
**Expressed as 100 percent glutaraldehyde.

gave appreciable resistance to perspiration, with an average loss in strength of only 25 percent as compared with 65 percent for the controls. It can be assumed that 0.75 percent of glutaraldehyde is the least amount that will produce significant perspiration resistance.

Results of dipping experiments using finished belly leathers and cut insoles are shown in Table II. The data for finished belly pieces present an average percent pickup of glutaraldehyde and a reduction of percent loss in tensile strength. The treated leathers clearly show an increased resistance to perspiration. The data also show that, although the average percent pickup of glutaraldehyde was approximately the same for both types of cut insoles, the buffed insoles showed a 40 percent increase in resistance, while the full grain insoles showed only about seven to eight percent increased resistance to perspiration. The insoles were obtained from two sources and the initial tensile strength of each was noticeably different. Both types of insoles had a shrinkage temperature of 79–80°C. before dipping and 82–83°C. after dipping.

## TABLE II

FINISHED BELLY PIECES AND CUT INSOLES DIPPED IN GLUTARALDEHYDE SOLUTION* AND SUBJECTED TO PERSPIRATION TEST

| Experiment | Percent Glutaraldehyde Pickup | Tensile Strength (PSI) | | | Average Percent Loss of Strength | |
|---|---|---|---|---|---|---|
| | | Control† | Test‡ | Untreated Leather | Control | Test |
| | | | Finished Belly Pieces | | | |
| 1** | 1.0 | 1420 | 1760 | | | |
| 2** | 1.3 | 850 | 2000 | 3730†† | 65.4 | 48.5 |
| 3** | 1.1 | 1590 | 1990 | | | |
| | | | Cut Insoles | | | |
| Buffed‡‡ | 1.20 | 760 | 1800 | 2590†† | 70.7 | 30.5 |
| Full Grain‡‡ | 1.29 | 1750 | 2050 | 3940†† | 55.6 | 47.7 |

*Dipped in ten times their weight of a solution containing 2.5 percent glutaraldehyde.
†Regular leather subjected to synthetic perspiration.
‡Dipped in glutaraldehyde and subjected to perspiration.
**Each consisting of eight to ten pieces measuring 4 x 12 inches.
††Leather which was not subjected to glutaraldehyde treatment, perspiration solution, or flexing. Figure is an average result of 20 test strips and was used to calculate average percent loss of strength.
‡‡Twenty insoles were used in each test.

The untreated leathers, both belly pieces and cut insoles, varied in tensile strength from 2100 to 4900 PSI. This variable quality, which is inherent in leather in general, accounted for the spread of results obtained. The center of the belly is the strongest and most uniform area, judging from measurements made on the strips cut from known areas of the bellies.

## DISCUSSION

Dipped insoles showed a negligible amount of shrinkage, the color was clear but slightly lightened, the surface was not spotted and thickness was not affected. The grain was minutely accentuated. In all dipping experiments there was a slight loss of water-soluble material, indicated by the light brown coloration of the solution. A sufficient number of test pieces were used to establish reliable results.

The perspiration treatment on the Clarke-Flaharty Insole Tester was conducted twice. After the first treatment all the leathers were slightly darkened. After the second treatment the control pieces were darkened and distorted and some appeared gelatinized. The strips treated with glutaraldehyde darkened but were not distorted or gelatinized.

The results indicated that the most feasible place to add glutaraldehyde to belly leather is in the oil wheel. This eliminated the need for dipping and insured a more uniform distribution, although the higher concentration of glutaraldehyde was somewhat irritating to the workers. The treatment amounts to a quick retannage without adding another step to the processing.

If dipping is preferred, a rocking motion or other agitation would help equilibrate the absorption and penetration of glutaraldehyde by the cut insoles. A dilute glutaraldehyde solution is desirable because it showed little visual effect, whereas a concentrated solution tended to produce a harsh cracky grain. There is no discomfort incurred in the dipping procedure.

In conclusion, it appears that glutaraldehyde used either in the oil wheel or by dipping does improve the resistance of insole leather to perspiration by as much as 40 percent. The leather does not harden or stiffen as untreated leather does when exposed to excessive perspiration.

## ACKNOWLEDGMENT

The authors acknowledge with thanks the assistance of William E. Palm in making the physical tests and the co-operation of Armour Leather Company at Williamsport, Pennsylvania.

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